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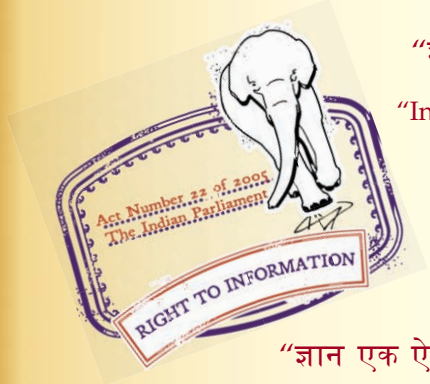

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SPECIFICATION FOR TRIMETHYLAMINE, TECHNICAL

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Indian Standard

SPECIFICATION FOR TRIMETHYLAMINE, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 22 November 1977, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Trimethylamine is used in the manufacture of epoxy resins, animal feeds, drugs and pharmaceuticals, ion-exchange resins, agricultural chemicals, etc. It also finds use in germicides, textiles, detergents and corrosion inhibitors, and as catalyst for reaction polymerization. It is also widely used as plant growth controller and solvent.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for trimethylamine, technical.

2. TYPES

2.1 The material shall be of the following types:

a) Type 1 — anhydrous,

b) Type 2 — 40 percent solution, and

c) Type 3 — 30 percent solution.

*Rules for rounding off numerical values (*revised*).

3. REQUIREMENTS

3.1 Description

3.1.1 *Type 1* — The material shall mainly consist of trimethylamine [$(CH_3)_3N$] and shall be in the form of gas or colourless liquid under pressure, and shall possess characteristic odour of fish.

3.1.2 *Types 2 and 3* — The material shall be in liquid form and shall possess characteristic odour of fish.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Appendix A.

TABLE 1 REQUIREMENTS FOR TRIMETHYLAMINE, TECHNICAL

Sl No.	Characteristic	Requirement			Method of Test, Ref to
		Type 1	Type 2	Type 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Trimethylamine content, percent by mass, *Min*	99·5	40	30·0	Appendix A
ii)	Monomethylamine, percent by mass, *Max*	0·30	0·1	0·1	
iii)	Dimethylamine, percent by mass, *Max*	0·30	0·1	0·1	
iv)	Moisture, percent by mass, *Max*	0·5	—	—	
v)	Ammonia, percent by mass, *Max*	0·01	Traces	Traces	

4. PRECAUTIONS IN HANDLING

4.1 The material being flammable and corrosive, necessary precautions shall be taken while handling.

5. PACKING AND MARKING

5.1 Packing — The gaseous material shall be filled in gas cylinders under pressure. The material in the solution form shall be packed in mild steel drums.

5.1.1 Necessary safeguard against the risk arising from the storage and handling of this material shall be provided and precautions shall be taken at all times to prevent accident by fire and explosion.

5.1.2 All containers for storage and transport of the material shall, in addition, comply with the requirements of the Red Tariff issued by Indian Railway Conference Association, and the requirements laid down from time to time by the Chief Inspector of Explosives, Government of India, for packing, storage and transit of flammable liquids.

5.2 Marking

5.2.1 The containers shall be securely closed and shall bear legibly and indelibly the following information:

a) Name and type of the material;

b) Net mass of the material;

c) Name of the manufacturer and his recognized trade-mark, if any; and

d) Lot or batch number in code or otherwise.

5.2.2 All containers in which the material is stored or transported shall be prominently and clearly marked with the legend:

DANGER ! EXTREMELY FLAMMABLE HAZARDOUS LIQUID AND VAPOUR UNDER PRESSURE. LIQUID CAUSES BURNS. VAPOUR EXTREMELY IRRITATING.

5.2.3 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

6. SAMPLING

6.1 Representative samples of the material shall be drawn and their conformity to the standard judged as prescribed in Appendix B.

APPENDIX A

(*Clause* 3.2, and *Table* 1)

METHODS OF TEST FOR TRIMETHYLAMINE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. ANALYSIS OF TRIMETHYLAMINE, ANHYDROUS

A-2.0 General — Anhydrous methylamine is analyzed gas-chromatographically by injecting a known volume of the gas and calculating the percentages by mass by the method of area normalization with response factors determined by injecting a standard mixture.

A-2.1 Apparatus

A-2.1.1 *Gas Chromatograph* — with thermal conductivity detector (hot wire type).

A-2.1.2 *Potentiometric Strip-Chart Recorder* — full scale deflection 1 mV.

A-2.1.3 *Column* — of stainless steel or glass; 185 cm long, 4 mm in internal diameter and 6 mm in external diameter; packed with *Porapak Q*; (500-842 μm) coated with 10 percent (*m/m*) of a mixture of 8·9 percent (*m/m*) tetra-ethylene pentamine and 1·1 percent (*m/m*) potassium hydroxide.

A-2.1.4 *Syringes* — 2-ml and 1 μl.

A-2.1.5 *Sampling Bomb* — of stainless steel, 2·4 m long and 3·75 cm in diameter, fitted with needle valves at both ends with 6 mm N.P.T. The bomb should be able to withstand pressure up to 18 kg/cm^2.

A-2.1.6 *Electric Oven* — provided with thermostat fitted inside with a stainless steel coil of 3 mm diameter with ends protruding out through holes on both the side walls.

A-2.2 Test Substances

A-2.2.1 *Ammonia, Monomethylamine, Dimethylamine, Trimethylamine, Methanol and Water*

*Specification for water for general laboratory use (*second revision*).

A-2.2.2 *Standard Mixture* — A standard mixture of ammonia, monomethylamine, dimethylamine, trimethylamine, methanol and water is prepared on *m/m* basis, taking care that the total vapour pressure of the mixture does not exceed 1 kg/cm^2.

A-2.3 Procedure

A-2.3.1 *Operating Parameters of Gas Chromatograph*

A-2.3.1.1 *Column oven temperature* — 90°C (isothermal).

A-2.3.1.2 *Injection port temperature* — 150°C.

A-2.3.1.3 *Detector block temperature* — 150°C.

A-2.3.1.4 *Carrier gas* — Hydrogen with 50 ml/min flow rate.

A-2.3.1.5 *Delivery pressure of carrier gas* — 1·4 kg/cm^2.

A-2.3.1.6 *Bridge current* — 200 mA.

A-2.3.1.7 *Chart speed* — 30 cm/h.

A-2.3.2 *Determination of Response Factors* — Check and adjust the chromatograph. Inject 1 μl of the standard mixture with the help of the hypodermic syringe. By suitably manipulating, the attenuator switch, record all the peaks on the chart. Measure the area of all the individual peaks. Calculate the response factors as follows:

a) Corresponding to each peak of the standard mixture, determine the amount of area produced by mass percent of the component.

b) Select one peak (monomethylamine) as a reference. Set its response factor (area by mass percent) equal to 1 and express all other response factors relative to it.

A-2.3.3 *Sample Injection* — Connect the bomb containing the sample under pressure vertically to one end of the heated coil in the oven with swage lock metallic fittings. Connect the other end (exit end) of the coil with rubber tubing to a bubbler, half-filled with water, the exit end of the bubbler being connected to a long rubber tubing which is taken outside the room as a vent. Now open slowly the bottom valve of the sample bomb. The sample would get immediately vaporized as it passes through the heated coil kept at 150°C. Take the vapour coming out through the outlet end of the coil in the 2-ml syringe by piercing the needle through the connecting rubber tubing. Inject 1 ml of the gaseous sample into the chromatograph and, by suitably manipulating the attenuator, record all the peaks on the chart.

Measure the areas of all the peaks and calculate the percentage (*m/m*) of each component as in **A-2.4**.

A-2.4 Calculation

A-2.4.1 For each peak, divide the measured area by the corresponding response factor to obtain the corrected area.

A-2.4.2 Add up all the corrected areas and calculate each corrected area as a percent of the total corrected area. These percentages are the mass percentages of the components in the sample.

A-3. ANALYSIS OF TRIMETHYLAMINE SOLUTION

A-3.0 General — The strength of a particular methylamine in solution is determined in two stages. In the first stage, total alkalinity of the solution is determined by titrating against standard acid and the alkalinity expressed in terms of percent of the particular amine. In the second stage, the impurities in the solution are determined gas chromatographically and each impurity (ammonia and amines) is expressed as the corresponding amine of which the solution is made. The sum total of these impurities is then subtracted from the total amine content to get the percent of the trimethylamine.

A-3.1 Determination of Total Alkalinity (as Trimethylamine)

A-3.1.1 *Reagents*

A-3.1.1.1 *Standard hydrochloric acid* — 1 N.

A-3.1.1.2 *Phenolphthalein indicator solution* — Dissolve 0·1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

A-3.1.2 *Procedure* — Take about 100 ml of water in a 250-ml conical flask and weigh. Pipette 10 ml of sample into it, keeping the tip of the pipette dipped in water while releasing the sample. Weigh it again. The difference of mass gives the mass of the sample. Titrate the contents with standard hydrochloric acid using phenolphthalein solution as indicator.

A-3.1.3 *Calculation*

$$\text{Total alkalinity } (X_1) \text{ (as trimethylamine)} = \frac{V \times N \times 0{\cdot}059 \times 100}{M}$$

where

V = volume in ml of standard hydrochloric acid used in the titration with the sample solution,

N = normality of standard hydrochloric acid, and

M = mass in g of the sample taken for the test.

A-3.2 Determination of Impurities by Gas Chromatographic Method

A-3.2.1 *Apparatus*

A-3.2.1.1 *Gas chromatograph* — with thermal conductivity detector (hot wire type).

A-3.2.1.2 *Potentiometric strip-chart recorder* — full scale deflection 1 mV.

A-3.2.1.3 *Column* — of stainless steel or glass; 185 cm long, 4 mm in internal diameter and 6 mm in external diameter; packed with *Porapak Q* (500-842 microns) coated with a 10 percent mixture of 8·9 percent (*m/m*) tetra-ethylene pentamine and 1·1 percent (*m/m*) potassium hydroxide.

A-3.2.1.4 *Syringe* — 10 µl.

A-3.2.2 *Test Substances*

A-3.2.2.1 *Ammonia, monomethylamine, dimethylamine, trimethylamine, methanol and water*

A-3.2.2.2 *Standard mixture* — A standard mixture of ammonia, monomethylamine, dimethylamine, trimethylamine, methanol and water is prepared on *m/m* basis, preferably, in concentration similar to that expected in the sample, taking care that the total vapour pressure of the mixture does not exceed 1 kg/cm^2.

A-3.2.3 *Procedure*

A-3.2.3.1 *Operating parameters of gas chromatograph*

a) *Column temperature* — 90°C.

b) *Injection port temperature* — 150°C.

c) *Detector block temperature* — 150°C.

d) *Carrier gas* — hydrogen, with 50 ml/min flow rate and 1·4 kg/cm^2 delivery pressure.

e) *Bridge current* — 200 mA.

f) *Chart speed* — 30 cm/h.

A-3.2.3.2 *Testing* — Check and adjust the gas chromatograph. Inject 1 µl of the standard mixture with the help of the syringe. By suitably manipulating the attenuator switch, record the peaks on the chart and measure the area of the individual peaks.

Under identical conditions, inject 1 µl of the sample and measure peak areas for all individual peaks as in the case of standard mixture.

A-3.2.3.3 *Elution order* — Elution order of the components is:

Ammonia, monomethylamine, dimethylamine, trimethylamine, methanol and water.

A-3.2.4 *Calculation*

A-3.2.4.1

$$P_s = \frac{A_s \times P_{std} \times S_1}{A_{std} \times S_2}$$

where

P_s = percent by mass of the component in the sample,

P_{std} = percent by mass of the component in the standard mixture,

A_s = area of the component in the case of sample,

A_{std} = area of the component in the case of standard mixture,

S_1 = attenuation used for the sample, and

S_2 = attenuation used for the standard mixture.

A-3.2.4.2 Trimethylamine content, percent by mass $= X_1 - (X_2 + X_3 + X_4)$

where

X_1 = total alkalinity (as trimethylamine), percent by mass (**A-3.1.3**);

X_2 = ammonia content (NH_3) in terms of trimethylamine, percent by mass, in the sample (**A-3.2.4.1**) = percent $NH_3 \times \frac{59}{17}$;

X_3 = monomethylamine content (MMA) in terms of trimethylamine, percent by mass, in the sample (**A-3.2.4.1**) = percent MMA $\times \frac{59}{31}$; and

X_4 = dimethylamine content (DMA) in terms of trimethylamine, percent by mass, in the sample (**A-3.2.4.1**) = percent DMA $\times \frac{59}{45}$.

APPENDIX B

(*Clause* 6.1)

SAMPLING OF TRIMETHYLAMINE, TECHNICAL

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.1 The sampling instrument shall be clean and dry.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.4 The samples shall be placed in suitable clean, dry and airtight metal or dark or amber glass containers on which the material has no action.

B-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.6 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling, and the month and year of manufacture of the material.

B-1.7 The samples shall be stored in the dark.

B-2. SAMPLING INSTRUMENT

B-2.1 The following forms of sampling instruments may be used:

a) Sampling bottle or can, for taking samples from tanks or drums; and

b) Sampling tube, for taking samples from bottles or small containers.

B-2.1.1 *Sampling Bottle or Can* — consists of a weighted glass or metal container with removable stopper or top to which is attached a light chain (*see* Fig. 1). The bottle or the can is fastened to a suitable pole. For taking a sample, the bottle or the can is lowered into the tank or drum to the required depth and the stopper is then removed by means of the chain.

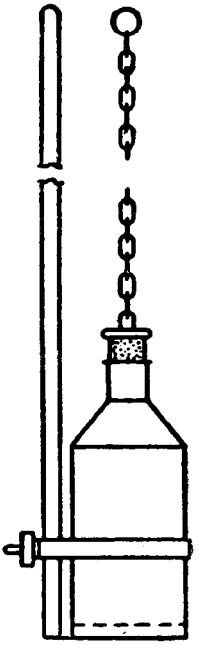

FIG. 1 SAMPLING BOTTLE OR CAN

B-2.1.2 *Sampling Tube* — made of metal or thick glass, is 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 2). The ends are conical and reach 5 to 10 mm diameter. Handling is facilitated by two rings at the upper end.

B-2.1.2.1 For small containers, the size of the sampling tube may be altered suitably.

B-3. SCALE OF SAMPLING

B-3.1 For Tanks and Drums — Each tank or drum shall be sampled separately.

B-3.2 For Bottles and Small Containers — Each lot shall be sampled separately.

B-3.2.1 *Lot* — In any consignment, all the containers of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

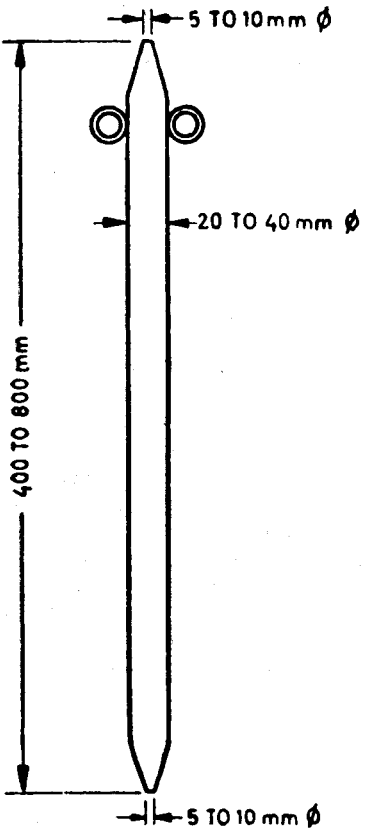

FIG. 2 SAMPLING TUBE

B-3.2.2 Tests shall be conducted on each lot separately for ascertaining its conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with Table 2.

B-3.2.3 The containers shall be chosen at random from the lot with the help of a suitable random number table. Reference may be made to IS : 4905-1968* for guidance for random selection procedures.

*Methods for random sampling.

TABLE 2 SCALE OF SAMPLING

(*Clause* B-3.2.2)

LOT SIZE	NO. OF CONTAINERS TO BE SELECTED
(1)	(2)
Up to 15	3
16 ,, 40	4
41 ,, 65	5
66 ,, 110	7
111 and above	10

NOTE — Where the size of the lot is three or less, all the containers shall be sampled.

B-4. COMPOSITE SAMPLE

B-4.1 As far as possible, samples from a tank or drum should be drawn during the operation of filling. In that case equal amounts of the material shall be collected at regular intervals so as to get a total of about 1 500 ml. Where it is not possible to take a sample during filling, the material shall be drawn from different positions and depths with the sampling bottle or can after thoroughly agitating the material so as to ensure a fair amount of homogeneity. The total amount of the material collected shall be thoroughly mixed and divided into 3 equal portions, one for the purchaser, another for the supplier and the third for the referee.

B-4.2 All the test samples shall be transferred to separate sample containers, sealed and labelled with full identification particulars. The referee test sample bearing the seal of both the purchaser and the supplier shall be kept at a place agreed to between the two and shall be used in case of a dispute.

B-4.3 Tests for the determination of all the requirements given in this specification shall be performed on the test sample obtained as in **B-4.1.**

B-5. CRITERIA FOR CONFORMITY

B-5.1 The lot shall be declared as conforming to this specification if all the test results satisfy the requirements prescribed under **3.**